MECHANOCHEMICAL TOLUENE NITRATION

Using shaker mill in Lab 221, York Center

STANDARD OPERATING PROCEDURE

Revised 02/09/2017

1. Wear appropriate PPE (e.g. goggles, gloves, laboratory coat, etc.)
2. Clean and air-dry the milling vials as per the mill manufacturer's instructions.
3. Gather all materials and supplies needed for the loading: reactants, milling balls, weighing dishes, syringe, spatula; and weighing balance.
4. Inside a fume hood, weigh desired amounts of MoO_3 and $NaNO_3$ in aluminum/ plastic weighing dishes and record the powder masses to the nearest 0.001 g.
5. Weigh the desired amount of milling balls and record their mass to the nearest 0.1 g.
6. Load MoO_3 and $NaNO_3$ into the milling vials.
7. Load the milling balls into the milling vials.
8. Using the syringe, add the desired volume of toluene into the milling vials and quickly cover the vials with lids. Screw on the vial cover rings as tightly as possible without using tools.
9. Place the vials into the vial holders inside the shaker mill and tighten the lock nuts.
10. Turn on additional air flow by opening the compressed air valve on the side of the fume hood.
11. Close and lock the shaker mill cover.
12. Turn on the power switch on the back wall of the shaker mill.
13. Set the desired milling time and turn on the mill.
14. When the mill stops at the end of the milling process open the mill cover and remove the vials.
15. Let the vials cool to room temperature for at least 5 minutes.
16. Prepare two containers (glass vial, beaker, etc.) with 15 ml ethyl acetate each.
17. Open one of the vials, add 15 ml of ethyl acetate, and replace the cover. Repeat for the second vial.
18. Place both vials into the vial holders inside the shaker mill, tighten the lock nuts, close the mill cover, and mill for 30 seconds.
19. Open one of the vials, remove the liquid using a plastic pipette, and collect it in a glass or polyethylene centrifuge tube. Do not use polystyrene centrifuge tubes. Repeat for the second vial.
20. Centrifuge the tubes for 5 minutes.
21. Remove the liquid fraction from the first tube using clean polyethylene pipette, and collect in a labeled sample vial. Repeat for the second centrifuge tube.
22. Save the solid fraction for future analysis, or discard from the centrifuge tubes in the appropriate waste container.
23. Clean the milling vials as per manufacturer's instructions.